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**COMPARISON OF SAMPLING PROBE AND
THERMAL DESORBER IN HAZARDOUS AIR
POLLUTANTS ON SITE (HAPSITE) EXTENDED
RANGE (ER) FOR ANALYSIS OF TOXIC ORGANIC
(TO)-15 COMPOUNDS**

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Interim Report

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14. ABSTRACT The Hazardous Air Pollutants on Site (HAPSITE), a portable Gas Chromatograph-Mass Spectrometer (GCMS), has been used to detect, identify, and quantify Volatile Organic Compounds (VOCs) from environmental samples, providing on-site analysis to aid in operational risk management. HAPSITE is equipped with a hand-held sampling probe in which an air sample is delivered into a concentrator, and the VOCs collected in the concentrator are transferred, separated, and identified in the GC-MS. An upgraded version, HAPSITE ER, has recently been introduced with additional sampling capability for solid phase micro extraction and Thermal Desorption (TD). To our knowledge, however, no study has yet evaluated the performance of the thermal desorber accommodated in HAPSITE ER. In this study, therefore, we analyzed EPA Method TO-15 compounds with two different sampling methods (probe and thermal desorber for TD tubes) in a HAPSITE ER, and compared their results against each other. A major finding was that the peak intensities of the TO-15 compounds, particularly those with high Boiling Point (BP), were substantially higher in the results obtained with the thermal desorber than in those with the sampling probe. The lower peak intensities of the compounds observed in the probe analysis are likely due to the condensation of the VOCs in the probe (transfer) line that is 6 feet long and maintained at 40°C as they are delivered from the probe to the concentrator, whereas the thermal desorber is directly connected to the HAPSITE (no transfer line is used), thereby eliminating the condensation of VOCs. In conclusion, our study suggests that for the analysis of VOCs with high up to 220°C, the use of TD tubes followed by desorption in the thermal desorber offered by the newer version of HAPSITE is recommended.					
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Comparison of sampling probe and thermal desorber in HAPSITE ER for analysis of TO-15 compounds

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Abstract

The Hazardous Air Pollutants on Site (HAPSITE), a portable gas chromatograph-mass spectrometer (GC-MS), has been used to detect, identify, and quantify unknown hazardous materials, providing on-site analysis to aid in operational risk management. HAPSITE is equipped with a hand-held sampling probe via which an air sample is delivered into a concentrator, and the volatile organic compounds (VOCs) collected in the concentrator are transferred, separated and identified in the GC-MS. An upgraded version HAPSITE ER ("extended range") has recently been introduced and has the desorption capability for solid phase microextraction fiber and thermal desorption (TD) sorbent tubes. To our knowledge, however, no study has yet to evaluate the thermal desorbers. In this study, therefore, we analyzed the TO-15 compounds with two different sampling methods (probe and thermal desorber for TD tubes) in a HAPSITE ER, and compared their results against each other. A major finding here was that the intensities of the Toxic Organic (TO)-15 compounds, particularly those with high boiling point (bp), were substantially higher in the results obtained with the thermal desorber than in those with the sampling probe. The lower intensities of the compounds observed in the probe analysis are likely due to the condensation of the VOCs in the probe (transfer) line that is 6 feet long and maintained at 40°C as they are delivered from the probe to the concentrator, whereas the thermal desorber is directly connected to the HAPSITE (no transfer line is used), thereby eliminating the condensation of VOCs. In conclusion, our study demonstrates that the thermal desorption capability offered by the newer version of HAPSITE allows the accurate analysis of VOCs with bp up to 200°C.

Introduction

The Inficon Hazardous Air Pollutants on Site (HAPSITE[®]), a portable gas chromatograph-mass spectrometer (GC-MS), has been used to detect, identify, and quantify unknown hazardous materials (e.g. chemical warfare agents [CWAs], volatile toxic industry chemicals, etc.) in an operational environment (Smith et al. 2004; Sekiguchi et al. 2006; Fair et al. 2009; 2010; Gorder and Dettenmaier 2011; Johnston et al. 2013), providing on-site analysis to aid in operational risk management. HAPSITE is equipped with a hand-held sampling probe via which an air sample is delivered into a concentrator in the HAPSITE system. The volatile organic compounds (VOCs) collected in the concentrator are transferred and separated through a GC column. The GC effluents then pass through a membrane maintained at 80°C, where volatile analytes move to the MS while inorganic gases (e.g. nitrogen and oxygen) are discarded (Sekiguchi et al. 2006). A quadrupole mass spectrometric detector is operated under vacuum provided by a nonevaporative getter (NEG) and an ion sputter pump (Smith 2012).

While the probe method allows near real time analysis of an air sample, it limits the volume of the sample collected, i.e. the sensitivity. In addition, VOCs with high boiling point (bp) are more likely condensed in the probe (transfer) line that is 6 feet long and maintained at 40°C, when they are delivered from the probe to the concentrator. An upgraded version HAPSITE ER (“extended range”) has recently been introduced and has many advantages over the previous models. In particular, besides the sampling probe, the newer version can accommodate a solid phase microextraction (SPME) fiber desorber or a thermal desorber for a thermal desorption (TD) sorbent tube so that a SPME fiber or a TD tube can be used to collect and concentrate samples externally, thereby providing an enhanced analytical sensitivity. Since the desorbers are directly connected to HAPSITE ER (no transfer line is used), furthermore, the condensation of VOCs with high bp in the probe line that possibly occurs when the sampling probe is used can be minimized. To our knowledge, however, no study has yet to evaluate the thermal desorbers. In this study, therefore, we analyzed the TO-15 compounds with two different sampling methods (probe and thermal desorber for TD tubes) in a HAPSITE ER, and compared their results against each other.

Materials and methods

Thermal desorption sorbent tubes

Stainless steel (SS) TD tubes containing a single component sorbent Tenax[®] TA purchased from Markes International (South Wales, UK) were used in this study. All tubes were conditioned prior to use based on the manufacturer's instruction.

Preparation of TO-15 compounds in a bag

To prepare 20 ppbv TO-15 compounds in a 5 L ALTEF polypropylene bag (Jensen Inert Products, Coral Springs, FL, USA), 100 mL was taken from a cylinder of the TO-15 65 component mix (1 ppm concentration) purchased from Restek (Bellefonte, PA, USA) using a 100 mL gas-tight syringe and then spiked into the bag containing full of nitrogen. The bag was left overnight for equilibration prior to sampling. Then, 100 mL was taken from the bag with the HAPSITE sampling probe or transferred to a Tenax sorbent tube with a 100 mL gas-tight syringe for the thermal desorber analysis. The sampling and analysis of the TO-15 compounds were performed 3 times with each sampling method (probe and thermal desorber) in the HAPSITE.

HAPSITE

An HAPSITE[®] ER system obtained from Inficon (East Syracuse, NY, USA) was used for analysis of the TO-15 mix in this study. A non-polar column (100% polydimethylsiloxane; 15 m × 0.25 mm ID × 1.0µm df) was equipped into the HAPSITE. For both probe and thermal desorber analyses, the temperatures of membrane, valve oven and heated lines were 80, 70 and 70°C, respectively. The GC temperature program and parameters in the mass spectrometer were identical as well. The GC temperature program started at 50°C for 2 min, increased at 3°C/min to 80°C, at 12°C/min to 120°C, and at 26°C/min to 200°C where the final temperature was held for 5.6 min. The GC analysis time was 24 min. Nitrogen was used as the carrier gas at a constant pressure of around 85 kPa. The mass spectrometer was operated in the electron impact ionization mode at 70 eV. The mass scan range was m/z 41 to m/z 300, and the scan time was 0.78 sec. The only difference between probe and thermal desorber methods was that the TO-15 mix captured by the probe was delivered to the concentrator at 40°C, whereas the mix adsorbed in a Tenax tube was desorbed in the thermal desorber at 330°C for 10 min and then delivered to

the concentrator. Note that the actual desorption temperature of the sorbent tube does not reach to the set temperature (This issue will be addressed in a forthcoming paper). The HAPSITE ER injects known volumes of internal standards 1,3,5-tris(trifluoromethyl)benzene [TRIS] and bromopentafluorobenzene [BPFB] (10.7 ppm and 5.5 ppm, respectively) for each analysis from the internal standard canister purchased from Inficon.

Results and Discussion

Figure 1 shows the total ion chromatograms of the TO-15 compounds obtained with probe and thermal desorber methods in HAPSITE ER. Forty nine TO-15 compounds were detected with both sampling methods and no compound detected exclusively with one method was observed. The names, ions for quantification, retention times, boiling points and intensities for the compounds are listed in Table 1. We noticed several differences in the chromatograms obtained with the different methods. First, the retention time for each TO-15 compound differed slightly. Although the GC temperature program was identical between the two methods, the TO-15 compounds eluted up to 5 seconds later in the thermal desorber analysis than in the probe analysis (Figure 1). It is not clear why the difference occurred. Second, the intensities of the internal standards TRIS and BPFB were much higher in the chromatograms obtained with the thermal desorber than in those with the sampling probe (Figure 1). In the probe method, the standards are injected during the line purge event that occurs prior to sampling and that requires a purging with an air sample for 1 min (Personal communication with Inficon). Consequently, the internal standards are diluted with the sample even before the sample is actually being collected and delivered to the concentrator. On the other hand, no dilution of the standards occurs in the thermal desorber analysis since they are injected immediately after the thermal desorption of a TD tube is completed and the internal standards and the desorbed analytes are delivered together to the concentrator, thereby exhibiting the higher intensities in the chromatograms than in those obtained with the probe method. Finally, the intensities of the TO-15 compounds, particularly those with high bp, were substantially higher in the results obtained with the thermal desorber than in those with the sampling probe (Table 1 and Figure 2). Note that the concentrations of the TO-15 compounds taken by the probe and the Tenax TD tubes should be same (100 mL was taken from the 5 L bag containing 20 ppbv TO-15 mix each for the probe and

the TD tubes). The lower intensities of the compounds observed in the probe analysis are likely due to the condensation in the probe line that is 6 feet long and maintained at 40°C as they are delivered from the probe to the concentrator, as mentioned in Introduction. Also, it is noteworthy that the TO-15 compounds adsorbed in the Tenax tubes are almost completely desorbed in the thermal desorber and then delivered to the concentrator (Data not shown). In conclusion, our study demonstrates that the thermal desorption capability offered by the newer version of HAPSITE (ER) allows the accurate analysis of VOCs with bp up to 200°C.

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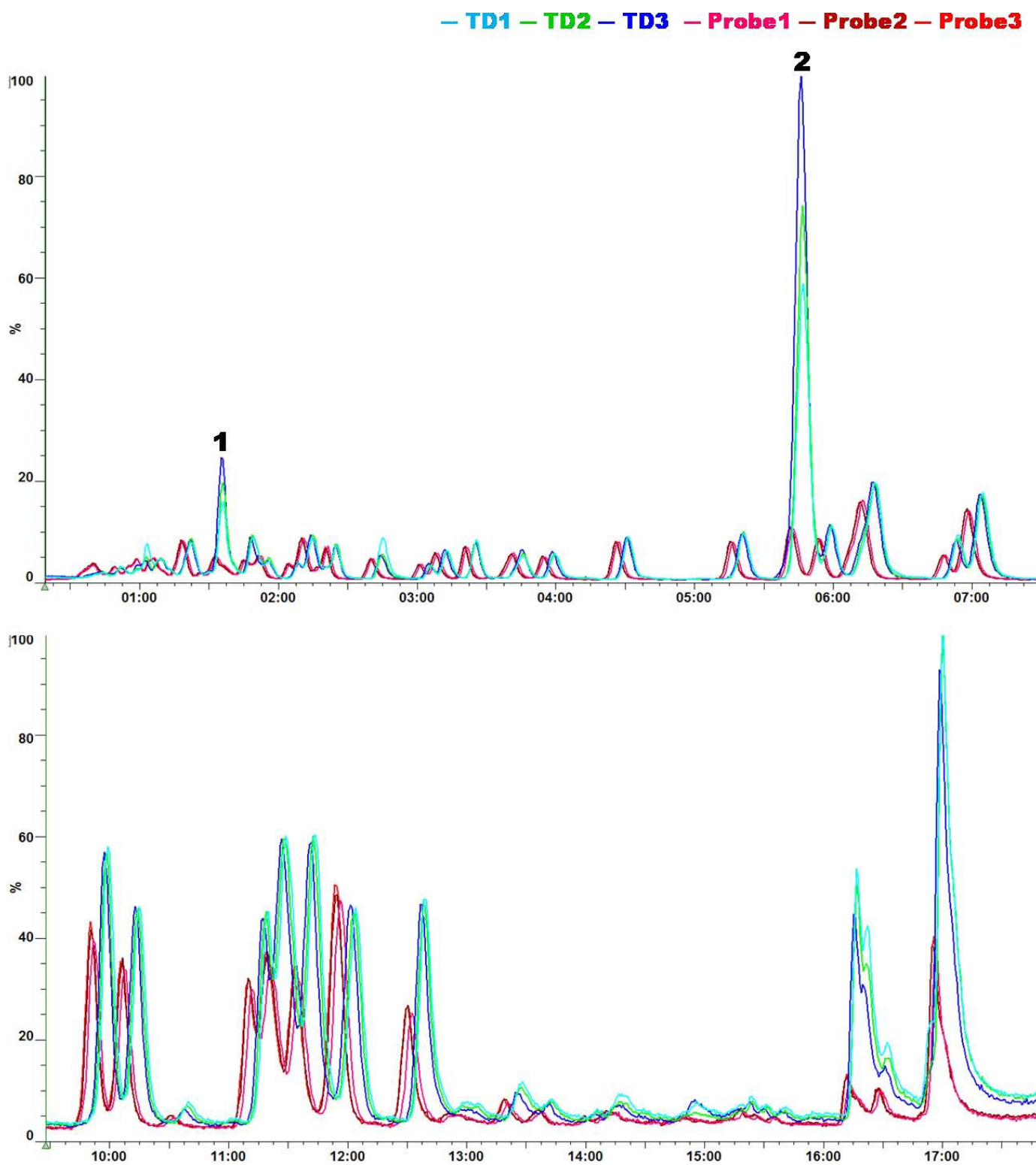


Figure 1. The total ion chromatograms of the TO-15 compounds analyzed with probe and thermal desorber methods in HAPSITE ER. The y-axis indicates relative intensity and the x-axis indicates

retention time in minutes. All chromatograms in each group are overlaid and designated by a different color. TRIS (1) and BPFB (2) are internal standards.

Table 1. TO-15 compounds detected in HAPSITE ER and their ions for quantification, retention times, boiling points and intensities obtained by different sampling methods.

Analyte Name	Q-Ion	RT	bp (°C)	Intensity of Q-ion @ 20 ppb in Taylor							
				TD				Probe			
				1	2	3	Average	1	2	3	Average
Acetone	58	0.80	57	201,000	146,000	139,000	162,000	126,000	117,000	124,000	122,333
Isopropanol	45	0.84	83	77,200	83,500	76,500	79,067	90,200	107,000	98,700	98,633
Trichloromonofluoromethane	101	0.83	24	82,700	96,800	77,100	85,533	169,000	164,000	161,000	164,667
1,1-Dichloroethene	61	0.92	32	186,000	199,000	189,000	191,333	211,000	210,000	200,000	207,000
1,1,2-Trichloro-1,2,2-trifluoroethane	151	0.97	48	12,900	12,900	12,200	12,667	55,500	61,100	53,600	56,733
Carbon disulfide	76	0.99	46	2,310,000	1,190,000	980,000	1,493,333	898,000	927,000	880,000	901,667
(E)-1,2-Dichloroethene	61	1.06	49	363,000	403,000	364,000	376,667	396,000	411,000	384,000	397,000
1,1-Dichloroethane	63	1.10	57	357,000	360,000	384,000	367,000	396,000	370,000	358,000	374,667
2-Butanone (MEK)	72	1.18	80	111,000	97,600	101,000	103,200	106,000	102,000	98,500	102,167
(Z)-1,2-Dichloroethene	61	1.27	60	414,000	416,000	411,000	413,667	423,000	420,000	397,000	413,333
Ethyl acetate	88	1.31	77	35,800	37,800	30,800	34,800	40,400	35,400	34,700	36,833
Hexane	57	1.31	69	423,000	478,000	464,000	455,000	479,000	462,000	461,000	467,333
Chloroform	83	1.34	61	485,000	503,000	496,000	494,667	499,000	519,000	513,000	510,333
Tetrahydrofuran	72	1.46	66	92,600	106,000	94,400	97,667	95,400	98,700	94,100	96,067
1,3,5-Tris(trifluoromethyl)benzene (TRIS; an IS)	213	1.56		799,000	965,000	1,380,000	1,048,000	184,000	207,000	181,000	190,667
1,2-Dichloroethane	62	1.54	84	507,000	528,000	501,000	512,000	442,000	461,000	469,000	457,333
1,1,1-Trichloroethane	97	1.61	74	188,000	219,000	198,000	201,667	256,000	264,000	252,000	257,333
Benzene	78	1.76	80	1,890,000	2,040,000	1,880,000	1,936,667	857,000	870,000	762,000	829,667
Carbon Tetrachloride	117	1.81	77	159,000	219,000	197,000	191,667	257,000	259,000	247,000	254,333
Cyclohexane	84	1.88	81	321,000	407,000	351,000	359,667	452,000	481,000	435,000	456,000
1,2-Dichloropropane	63	2.08	96	310,000	307,000	305,000	307,333	285,000	306,000	280,000	290,333
Bromodichloromethane	83	2.17	90	601,000	623,000	660,000	628,000	674,000	700,000	641,000	671,667
1,4-dioxane	88	2.20	101	154,000	160,000	137,000	150,333	105,000	119,000	113,000	112,333
Trichloroethylene	130	2.20	87	412,000	437,000	381,000	410,000	385,000	364,000	366,000	371,667
Heptane	71	2.36	99	516,000	548,000	487,000	517,000	483,000	478,000	490,000	483,667
(Z)-1,3-Dichloro-1-propene	75	2.67	104	559,000	573,000	646,000	592,667	618,000	575,000	555,000	582,667
Methyl Isobutyl Ketone	43	2.68	118	1,060,000	455,000	437,000	650,667	319,000	336,000	329,000	328,000
(E)-1,3-Dichloro-1-propene	75	3.04	112	560,000	592,000	648,000	600,000	634,000	650,000	574,000	619,333
1,1,2-Trichloroethane	97	3.15	115	397,000	388,000	405,000	396,667	385,000	373,000	372,000	376,667
Toluene	91	3.37	111	1,380,000	1,310,000	1,260,000	1,316,667	1,140,000	1,130,000	1,130,000	1,133,333
Methyl Butyl Ketone (2-Hexanone)	43	3.64	128	294,000	287,000	298,000	293,000	294,000	305,000	327,000	308,667
Dibromochloromethane	129	3.70	120	465,000	553,000	625,000	547,667	582,000	551,000	529,000	554,000
1,2-Dibromoethane	107	3.92	133	847,000	892,000	938,000	892,333	755,000	771,000	788,000	771,333
Tetrachloroethylene	166	4.45	121	602,000	620,000	601,000	607,667	541,000	543,000	542,000	542,000
Chlorobenzene	112	5.28	131	1,190,000	1,260,000	1,190,000	1,213,333	1,070,000	1,070,000	1,020,000	1,053,333
Bromopentafluorobenzene (BPFB; an IS)	117	5.70		6,730,000	8,500,000	11,400,000	8,876,667	1,220,000	1,200,000	1,190,000	1,203,333
Ethylbenzene	91	5.91	136	1,960,000	1,960,000	1,880,000	1,933,333	1,580,000	1,610,000	1,590,000	1,593,333
p/m-Xylene	91	6.21	139	2,870,000	2,720,000	2,890,000	2,826,667	2,360,000	2,300,000	2,340,000	2,333,333
Tribromomethane	173	6.13	151	555,000	681,000	682,000	639,333	523,000	512,000	510,000	515,000
Styrene	104	6.81	145	1,160,000	1,100,000	1,050,000	1,103,333	667,000	676,000	661,000	668,000
o-Xylene	91	6.97	144	1,390,000	1,380,000	1,410,000	1,393,333	1,130,000	1,180,000	1,200,000	1,170,000
1,1,2,2-Tetrachloroethane	83	6.98	147	1,180,000	1,160,000	1,150,000	1,163,333	958,000	935,000	937,000	943,333
1-Ethyl-4-methylbenzene (4-Ethyltoluene)	105	9.87	162	2,000,000	1,950,000	1,890,000	1,946,667	1,340,000	1,430,000	1,450,000	1,406,667
1,3,5-Trimethylbenzene (Mesitylene)	105	10.12	165	1,310,000	1,270,000	1,250,000	1,276,667	951,000	999,000	992,000	980,667
1,2,3-Trimethylbenzene (Hemimellitene)	105	11.20	176	1,280,000	1,290,000	1,260,000	1,276,667	822,000	883,000	874,000	859,667
Benzyl chloride	126	11.44	179	105,000	114,000	114,000	111,000	68,700	75,100	87,000	76,933
1,3-Dichlorobenzene	146	11.36	173	1,010,000	1,040,000	997,000	1,015,667	566,000	610,000	627,000	601,000
1,4-Dichlorobenzene	146	11.59	174	1,130,000	1,110,000	1,110,000	1,116,667	577,000	660,000	650,000	629,000
1,2-Dichlorobenzene	146	12.54	181	881,000	843,000	856,000	860,000	446,000	477,000	475,000	466,000
1,3,5-Trichlorobenzene	180	16.21	208	715,000	688,000	597,000	666,667	126,000	134,000	163,000	141,000
1,1,2,3,4,4-Hexachloro-1,3-butadiene	225	16.96	220	612,000	636,000	592,000	613,333	235,000	235,000	261,000	243,667

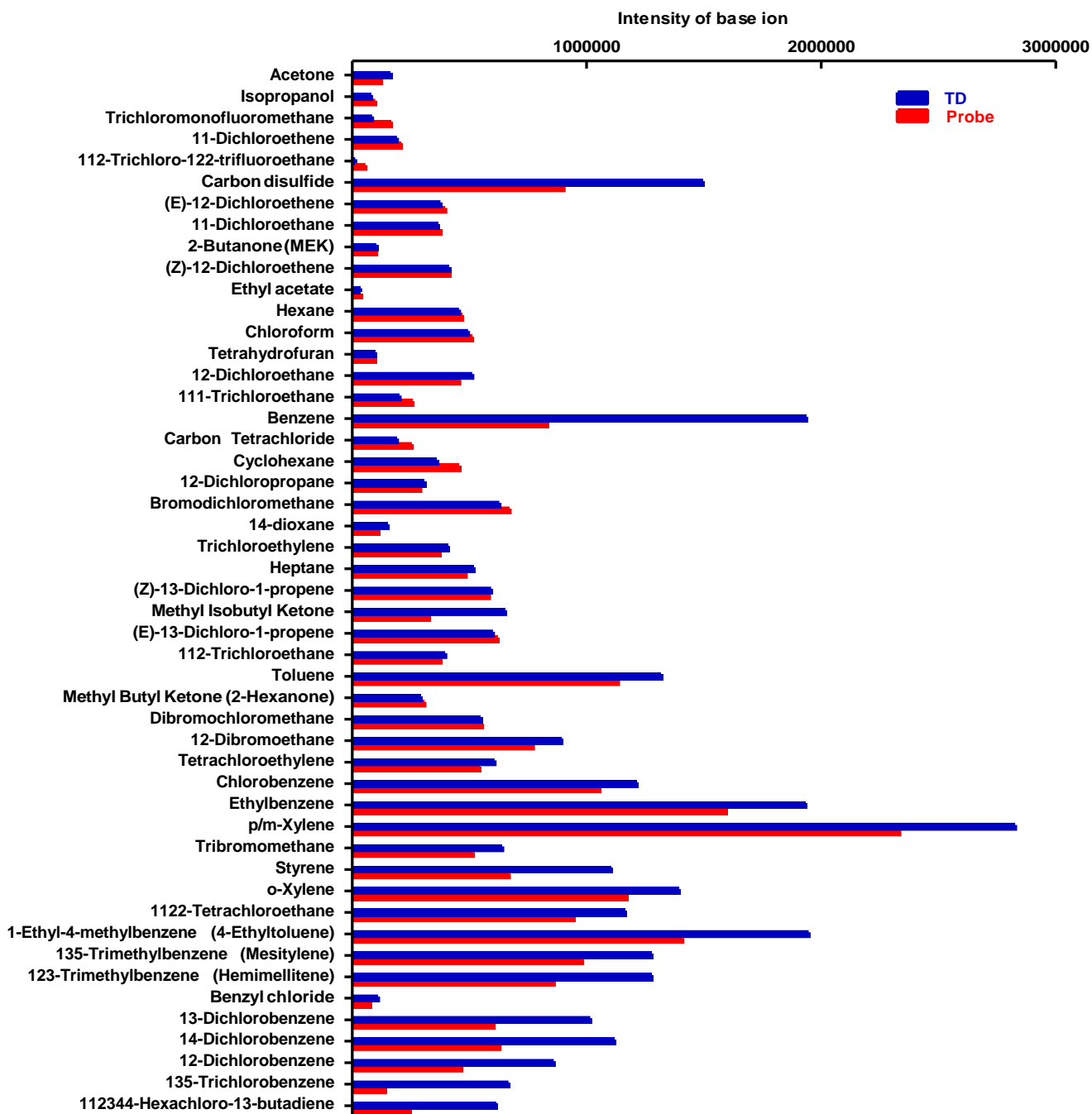


Figure 2. The averaged intensities of the TO-15 compounds obtained with different sampling methods in HAPSITE ER (probe vs. thermal desorber). N = 3 for each method.